

WEST**Freeform Search****Database:**

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L9 and width

Term:
Display: 50 **Documents in Display Format:** - **Starting with Number** 1

Generate: ☐ Hit List ☒ Hit Count ☐ Side by Side ☐ Image

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Cases

Search History
DATE: Tuesday, April 29, 2003 [Printable Copy](#) [Create Case](#)
Set Name **Query**
 side by side

Hit Count **Set Name**
 result set

DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ

<u>L10</u>	L9 and width	126	<u>L10</u>
<u>L9</u>	(l6 and l7)	411	<u>L9</u>
<u>L8</u>	l4 and l5	933	<u>L8</u>
<u>L7</u>	l2 and l4	836	<u>L7</u>
<u>L6</u>	l1 and l4	100163	<u>L6</u>
<u>L5</u>	?00nm or ?00nanometer 160-900	12050	<u>L5</u>
<u>L4</u>	silica or (silicon adj dioxide) or SiO ₂	505831	<u>L4</u>
<u>L3</u>	silica or (silicon adj dioxide) or SO ₂	484479	<u>L3</u>
<u>L2</u>	?0nm or ?0nanometer (10-90)	6826	<u>L2</u>
<u>L1</u>	\$1nm or \$1nanometer	320098	<u>L1</u>

Please return
these pages

Application/Control Number: 09/509,397

Page 2

Art Unit: 1711

DETAILED ACTION

1. A response (paper no. 12) has been filed on Feb. 11, 2002.
2. Claims under examination are 24-46.
- 3. Examiner thanks the applicant for pointing out an inadvertent error in earlier office action (paper no.9), items 3 and 5 wherein the correct words should have been "claims 24-46" and "claims 24-31, 34-37 and 39-46" ~~response~~ resp.
4. Rejection of claims 28, 29, 41, and 45 under 35 USC 112, second paragraph (see item 1, in office action, paper no.9) is maintained since the applicants' argument are not persuasive. Applicants' contention (on page 2 of above response paper no.12), that " the mol. wt. for a polymer generally means the wt. average of all molecules in the polymer sample "is not convincing. Word "salt" in claims 29 and 45 is generic and very broad and fails to convey which salts are encompassed by scope of these claims.
5. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.
6. Claims 24-26 are rejected under 35 U.S.C. 103(a) as being unpatentable over Assarsson et al (USP 3898143) in view of Hobbs (WO 96/04025).
- 7. Claims 24-31, 34-37 and 39-46 are rejected under 35 U.S.C. 102(b) as being anticipated by Assarsson et al (^{USP}~~USP~~ 3898143).

These rejections are incorporated here from prior office action paper no.9.

<u>Set Name</u> side by side	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u> result set
<i>DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ</i>			
<u>L16</u>	l1 near l2 [ab]	17	<u>L16</u>
<u>L15</u>	l1 near l2 [ti]	0	<u>L15</u>
<u>L14</u>	l1 near l2	140	<u>L14</u>
<u>L13</u>	l1 with l2	4521	<u>L13</u>
<u>L12</u>	l1 same l2	15847	<u>L12</u>
<u>L11</u>	l7 and l1	0	<u>L11</u>
<u>L10</u>	l8 and l1	0	<u>L10</u>
<u>L9</u>	l2 near5 l3 near1 l4	0	<u>L9</u>
<u>L8</u>	l2 near5 l3 near2 l4	3	<u>L8</u>
<u>L7</u>	l2 near5 l3 near5 l4	5	<u>L7</u>
<u>L6</u>	l2 near5 l3 near l4	0	<u>L6</u>
<u>L5</u>	l2 near l3 near l4	0	<u>L5</u>
<u>L4</u>	?0nm or ?0nanometer	6826	<u>L4</u>
<u>L3</u>	?nm or ?nanometer	9488	<u>L3</u>
<u>L2</u>	(width or wide)	2153543	<u>L2</u>
<u>L1</u>	silica or silicon dioxide	454892	<u>L1</u>

END OF SEARCH HISTORY

Art Unit: 1711

1.136(a) will be calculated from the mailing date of the advisory action. In no event, however,

will the statutory period for reply expire later than SIX MONTHS from the mailing date of this

final action.

8. Any inquiry concerning this communication or earlier communications from the examiner

should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The examiner

can normally be reached on Monday-Friday from 9:30 a.m. to 6:00 p.m.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor,

James J. Seidleck, can be reached on (703) 308-2462. The fax phone number for the organization

where this application or proceeding is assigned is (703)872-9310/9311.

Any inquiry of a general nature or relating to the status of this application or proceeding

should be directed to the receptionist whose telephone number is (703) 308-0661.

Rajguru/sp

June 10, 2002

WEST

Your wildcard search against 10000 terms has yielded the results below.

Your result set for the last L# is incomplete.

The probable cause is use of unlimited truncation. Revise your search strategy to use limited truncation.

Generate Collection

Print

Colvin

Search Results - Record(s) 1 through 36 of 36 returned.

1. Document ID: US 5552457 A

L30: Entry 1 of 36

File: USPT

Sep 3, 1996

US-PAT-NO: 5552457

DOCUMENT-IDENTIFIER: US 5552457 A

TITLE: Process for making a carbon black/resin masterbatch

DATE-ISSUED: September 3, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Bull, Jr.; Charles L.	North Lawrence	OH		
Colvin; Howard A.	Tallmadge	OH		

US-CL-CURRENT: 523/351; 524/571

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	NWIC
Draw Desc	Image										

2. Document ID: US 5523351 A

L30: Entry 2 of 36

File: USPT

Jun 4, 1996

US-PAT-NO: 5523351

DOCUMENT-IDENTIFIER: US 5523351 A

TITLE: Compatibilized polymeric systems

DATE-ISSUED: June 4, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Colvin; Howard A.	Tallmadge	OH		
Alsamarraie; Muhanad A.	Stow	OH		
Parker; Dane K.	Massillon	OH		

US-CL-CURRENT: 525/124; 525/125, 525/179, 525/63, 525/64, 525/66, 525/67, 525/70,
525/71, 525/74, 525/931

Application/Control Number: 09555591

Page 4

Art Unit: 1711

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-0661.

UKRajguru:evh

8/15/021

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC
Draw Desc	Image										

3. Document ID: US 5310815 A

L30: Entry 3 of 36

File: USPT

May 10, 1994

US-PAT-NO: 5310815

DOCUMENT-IDENTIFIER: US 5310815 A

TITLE: Polymers derived from a conjugated diolefin, a vinyl-substituted aromatic compound, and olefinically unsaturated nitrile

DATE-ISSUED: May 10, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Senyek; Michael L.	Tallmadge	OH		
Colvin; Howard A.	Tallmadge	OH		

US-CL-CURRENT: 525/329.3; 525/354

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC
Draw Desc	Image										

4. Document ID: US 5225479 A

L30: Entry 4 of 36

File: USPT

Jul 6, 1993

US-PAT-NO: 5225479

DOCUMENT-IDENTIFIER: US 5225479 A

TITLE: Polymers derived from a conjugated diolefin, a vinyl-substituted aromatic compound, and olefinically unsaturated nitrile

DATE-ISSUED: July 6, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Senyek; Michael L.	Tallmadge	OH		
Colvin; Howard A.	Tallmadge	OH		

US-CL-CURRENT: 524/526; 525/233, 525/234

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw Desc	Image									

5. Document ID: US 5191083 A

L30: Entry 5 of 36

File: USPT

Mar 2, 1993

US-PAT-NO: 5191083

DOCUMENT-IDENTIFIER: US 5191083 A



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Printer: cp3_4c07_gbgnptr
Date: 08/08/01
Time: 18:40:08

Document Listing

Document	Selected Pages	Page Range
US004655435	6	1 - 6
Total (1)	6	-

TITLE: Functionalized monomers from 1-(1-isocyanato-1-methlethyl)-3 or 4-(1-methylethenyl) benzene

DATE-ISSUED: March 2, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Akron	OH		
Cottman; Kirkwood S.	Akron	OH		
Parker; Dane K.	Massillon	OH		

US-CL-CURRENT: 546/222; 560/27, 560/29, 560/32

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWIC

6. Document ID: US 5173557 A

L30: Entry 6 of 36

File: USPT

Dec 22, 1992

US-PAT-NO: 5173557

DOCUMENT-IDENTIFIER: US 5173557 A

TITLE: Crosslinkable rubber composition

DATE-ISSUED: December 22, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parker; Dane K.	Massillon	OH		
Weinstein; Arthur H.	Hudson	OH		
<u>Colvin; Howard A.</u>	Tallmadge	OH		

US-CL-CURRENT: 526/301; 526/312, 548/530, 548/537

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWIC

7. Document ID: US 5043455 A

L30: Entry 7 of 36

File: USPT

Aug 27, 1991

US-PAT-NO: 5043455

DOCUMENT-IDENTIFIER: US 5043455 A

TITLE: Crosslinkable rubber composition

DATE-ISSUED: August 27, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parker; Dane K.	Massillon	OH		
Weinstein; Arthur H.	Hudson	OH		
<u>Colvin; Howard A.</u>	Tallmadge	OH		

Q

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Printer: cp3_4c07_gbgmpt
Date: 09/22/02
Time: 18:39:49

Document Listing

Document	Selected Pages	Page Range
US005393823	8	1 - 8
Total (1)	8	-

US-CL-CURRENT: 548/537; 548/530

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KMIC

8. Document ID: US 4983684 A

L30: Entry 8 of 36

File: USPT

Jan 8, 1991

US-PAT-NO: 4983684

DOCUMENT-IDENTIFIER: US 4983684 A

TITLE: Crosslinkable rubber composition

DATE-ISSUED: January 8, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Weinstein; Arthur H.	Hudson	OH		
<u>Colvin; Howard A.</u>	Tallmadge	OH		
Parker; Dane K.	Massillon	OH		

US-CL-CURRENT: 525/329.1; 525/124, 525/283, 525/330.3, 525/330.5, 525/331.9, 525/332.5,
525/332.9, 525/333.3, 525/333.5, 525/343, 525/374, 525/375, 525/377, 525/379, 525/380,
525/384, 525/386

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KMIC

9. Document ID: US 4973787 A

L30: Entry 9 of 36

File: USPT

Nov 27, 1990

US-PAT-NO: 4973787

DOCUMENT-IDENTIFIER: US 4973787 A

TITLE: Process for the thermal dimerization of isoprene

DATE-ISSUED: November 27, 1990

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Tallmadge	OH		

US-CL-CURRENT: 585/508; 585/5

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KMIC

10. Document ID: US 4902775 A

L30: Entry 10 of 36

File: USPT

Feb 20, 1990

US-PAT-NO: 4902775

HPS Trailer Page
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Walk-Up Printing

UserID: Q

Printer: cp3_4c07_gbgmpttr

Summary

Document	Pages	Printed	Missed
US005393823	8	8	0
Total (1)	8	8	0

DOCUMENT-IDENTIFIER: US 4902775 A

TITLE: Rubber vulcanization agents of sulfur olefin adduct

DATE-ISSUED: February 20, 1990

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Colvin; Howard A.	Tallmadge	OH		
Bull, Jr.; Charles L.	Akron	OH		
Magnus; Fredrick L.	Mogadore	OH		

US-CL-CURRENT: 528/389

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KVMC

11. Document ID: US 4855362 A

L30: Entry 11 of 36

File: USPT

Aug 8, 1989

US-PAT-NO: 4855362

DOCUMENT-IDENTIFIER: US 4855362 A

TITLE: Polymer alloy

DATE-ISSUED: August 8, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Muse, Jr.; Joel	Kent	OH		
Colvin; Howard A.	Tallmadge	OH		

US-CL-CURRENT: 525/194; 525/192, 525/211, 525/232, 525/233, 525/237

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KVMC

12. Document ID: US 4853478 A

L30: Entry 12 of 36

File: USPT

Aug 1, 1989

US-PAT-NO: 4853478

DOCUMENT-IDENTIFIER: US 4853478 A

TITLE: Functionalized monomers from 1-(1-isocyanato-1-methylethyl)-3 or 4-(1-methylethenyl) benzene

DATE-ISSUED: August 1, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Colvin; Howard A.	Akron	OH		
Cottman; Kirkwood S.	Akron	OH		
Parker; Dane K.	Massillon	OH		

Q

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Printer: cp3_4c07_gbgmptr

Date: 09/22/02

Time: 18:36:50

Document Listing

Document	Selected Pages	Page Range
US005393823	8	1 - 8
Total (1)	8	-

US-CL-CURRENT: 560/32; 525/328.2, 526/288, 526/301, 526/302, 526/305, 526/313, 546/222,
558/240

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

13. Document ID: US 4812487 A

L30: Entry 13 of 36

File: USPT

Mar 14, 1989

US-PAT-NO: 4812487

DOCUMENT-IDENTIFIER: US 4812487 A

TITLE: Ames-negative diamine curative for polyurethanes

DATE-ISSUED: March 14, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Tallmadge	OH		

US-CL-CURRENT: 521/159; 521/163, 528/63, 528/75, 528/76, 528/80, 528/83

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

14. Document ID: US 4792589 A

L30: Entry 14 of 36

File: USPT

Dec 20, 1988

US-PAT-NO: 4792589

DOCUMENT-IDENTIFIER: US 4792589 A

TITLE: Rubber vulcanization agents of sulfur and olefin

DATE-ISSUED: December 20, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Tallmadge	OH		
Bull, Jr.; Charles L.	Akron	OH		

US-CL-CURRENT: 525/343; 525/348

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

15. Document ID: US 4755627 A

L30: Entry 15 of 36

File: USPT

Jul 5, 1988

US-PAT-NO: 4755627

HPS Trailer Page
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UserID: q

Printer: cp3_4c07_gbgndptr

Summary

Document	Pages	Printed	Missed
US004837245	9	9	0
Total (1)	9	9	0

DOCUMENT-IDENTIFIER: US 4755627 A

TITLE: Process for the synthesis and purification of diisopropenylbenzene

DATE-ISSUED: July 5, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Colvin; Howard A.	Akron	OH		
Fiedler; Ronald D.	Atwater	OH		
Muse, Jr.; Joel	Kent	OH		
Smith; Donald E.	Tallmadge	OH		

US-CL-CURRENT: 585/440; 585/254, 585/277, 585/315, 585/321

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWWC

16. Document ID: US 4739036 A

L30: Entry 16 of 36

File: USPT

Apr 19, 1988

US-PAT-NO: 4739036

DOCUMENT-IDENTIFIER: US 4739036 A

TITLE: Rubber vulcanization agents and methods for their preparation

DATE-ISSUED: April 19, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Colvin; Howard A.	Tallmadge	OH		
Bull, Jr.; Charles L.	Akron	OH		

US-CL-CURRENT: 528/389; 106/287.32

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWWC

17. Document ID: US 4714772 A

L30: Entry 17 of 36

File: USPT

Dec 22, 1987

US-PAT-NO: 4714772

DOCUMENT-IDENTIFIER: US 4714772 A

TITLE: Functionalized monomers from 1-(1-isocyanato-1-methylethyl)-3 or 4-(1-methylethenyl) benzene

DATE-ISSUED: December 22, 1987

INVENTOR-INFORMATION:

q

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Printer: cp3_4c07_gbgndptr
Date: 09/20/02
Time: 21:38:43

Document Listing

Document	Selected Pages	Page Range
US004837245	9	1 - 9
Total (1)	9	-

NAME	CITY	STATE	ZIP CODE	COUNTRY
Colvin; Howard A.	Akron	OH		
Cottman; Kirkwood S.	Akron	OH		
Parker; Dane K.	Massillon	OH		

US-CL-CURRENT: 558/240

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw	Desc	Image							

KWC

18. Document ID: US 4694057 A

L30: Entry 18 of 36

File: USPT

Sep 15, 1987

US-PAT-NO: 4694057

DOCUMENT-IDENTIFIER: US 4694057 A

TITLE: Preparation of polymers containing pendant isocyanate groups and derivatives thereof by emulsion copolymerization

DATE-ISSUED: September 15, 1987

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Smith; Robert A.	Uniontown	OH		
Parker; Dane K.	Massillon	OH		
Colvin; Howard A.	Akron	OH		
Weinstein; Arthur H.	Hudson	OH		
Patterson; Dennis B.	Akron	OH		

US-CL-CURRENT: 526/206; 526/223, 526/310

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw	Desc	Image							

KWC

19. Document ID: US 4604439 A

L30: Entry 19 of 36

File: USPT

Aug 5, 1986

US-PAT-NO: 4604439

DOCUMENT-IDENTIFIER: US 4604439 A

**** See image for Certificate of Correction ****

TITLE: Functionalized monomers from 1-(1-isocyanato-1-methylethyl)-3- or 4-(1-methylethenyl) benzene

DATE-ISSUED: August 5, 1986

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Colvin; Howard A.	Akron	OH		
Cottman; Kirkwood S.	Akron	OH		
Parker; Dane K.	Massillon	OH		

US-CL-CURRENT: 526/288; 526/301, 526/302

q

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Printer: cp3_4c07_gbgnptr

Date: 09/20/02

Time: 18:19:20

Document Listing

Document	Selected Pages	Page Range
US005418293	42	1 - 42
Total (1)	42	-

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWWC

20. Document ID: US 4538002 A

L30: Entry 20 of 36

File: USPT

Aug 27, 1985

US-PAT-NO: 4538002

DOCUMENT-IDENTIFIER: US 4538002 A

** See image for Certificate of Correction **

TITLE: Process for the production of hydroxyanisole and alkylated hydroxyanisoles

DATE-ISSUED: August 27, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Akron	OH		
Muse, Jr.; Joel	Kent	OH		
Hollingshead; William S.	Cuyahoga Falls	OH		

US-CL-CURRENT: 568/650; 568/658, 568/782

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWWC

21. Document ID: US 4532011 A

L30: Entry 21 of 36

File: USPT

Jul 30, 1985

US-PAT-NO: 4532011

DOCUMENT-IDENTIFIER: US 4532011 A

TITLE: Inhibiting polymerization of vinylaromatic monomers

DATE-ISSUED: July 30, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Akron	OH		

US-CL-CURRENT: 203/9; 203/60, 203/8, 585/5, 585/807

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWWC

22. Document ID: US 4528413 A

L30: Entry 22 of 36

File: USPT

Jul 9, 1985

US-PAT-NO: 4528413

DOCUMENT-IDENTIFIER: US 4528413 A

HPS Trailer Page
for

Walk-Up Printing

UserID: q

Printer: cp3_4c07_gbgmpttr

Summary

Document	Pages	Printed	Missed
US004562237	9	9	0
Total (1)	9	9	0

TITLE: Process for the synthesis and purification of diisopropenylbenzene

DATE-ISSUED: July 9, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Akron	OH		
Fiedler; Ronald D.	Atwater	OH		
Muse, Jr.; Joel	Kent	OH		
Smith; Donald E.	Tallmadge	OH		

US-CL-CURRENT: 585/440; 585/254, 585/277, 585/315, 585/321

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Drawn Desc	Image								

KIMC

23. Document ID: US 4431849 A

L30: Entry 23 of 36

File: USPT

Feb 14, 1984

US-PAT-NO: 4431849

DOCUMENT-IDENTIFIER: US 4431849 A

TITLE: Process for preparing a methyl phenol

DATE-ISSUED: February 14, 1984

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Akron	OH		

US-CL-CURRENT: 568/799; 568/771, 568/772, 568/798, 568/802

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Drawn Desc	Image								

KIMC

24. Document ID: US 4390741 A

L30: Entry 24 of 36

File: USPT

Jun 28, 1983

US-PAT-NO: 4390741

DOCUMENT-IDENTIFIER: US 4390741 A

TITLE: Process for the purification of diisopropenylbenzene

DATE-ISSUED: June 28, 1983

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Colvin; Howard A.</u>	Akron	OH		
Muse, Jr.; Joel	Kent	OH		

US-CL-CURRENT: 585/258; 585/266, 585/271, 585/804, 585/807

q

Printed by HPS Server
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Printer: cp3_4c07_gbgmptr

Date: 09/20/02

Time: 17:56:16

Document Listing

Document	Selected Pages	Page Range
US004562237	9	1 - 9
Total (1)	9	-

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KVMC

25. Document ID: JP 2000017021 A

L30: Entry 25 of 36

File: JPAB

Jan 18, 2000

PUB-NO: JP02000017021A

DOCUMENT-IDENTIFIER: JP 2000017021 A

TITLE: STYRENE-BUTADIENE RUBBER PRODUCED BY EMULSION POLYMERIZATION

PUBN-DATE: January 18, 2000

INVENTOR-INFORMATION:

NAME

COUNTRY

COLVIN, HOWARD A

SENYEK, MICHAEL L

INT-CL (IPC): C08 F 212/08; B60 C 1/00; C08 F 2/00; C08 F 2/22; C08 L 9/06

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KVMC

26. Document ID: JP 11315167 A

L30: Entry 26 of 36

File: JPAB

Nov 16, 1999

PUB-NO: JP411315167A

DOCUMENT-IDENTIFIER: JP 11315167 A

TITLE: ELASTOMER BLEND AND ITS USE IN TIRE

PUBN-DATE: November 16, 1999

INVENTOR-INFORMATION:

NAME

COUNTRY

SANDSTROM, PAUL HARRY

BLOK, EDWARD JOHN

ZANZIG, DAVID JOHN

COLVIN, HOWARD A

SENYEK, MICHAEL L

INT-CL (IPC): C08 L 9/00; B29 C 33/02; B29 C 35/02; B29 D 30/06; B60 C 1/00; C08 L 9/08

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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KVMC

27. Document ID: JP 11263881 A

L30: Entry 27 of 36

File: JPAB

Sep 28, 1999

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UserID: q

Printer: cp3_4c07_gbgmptr

Summary

Document	Pages	Printed	Missed
US006040009	39	39	0
US005393823	8	8	0
US004972027	22	22	0
US004923945	16	16	0
Total (4)	85	85	0

PUB-NO: JP411263881A
DOCUMENT-IDENTIFIER: JP 11263881 A
TITLE: RUBBER BLEND FOR TIRE TREAD COMPOUND

PUBN-DATE: September 28, 1999

INVENTOR-INFORMATION:

NAME

COUNTRY

COLVIN, HOWARD A

SENYEK, MICHAEL L

FUTAMURA, SHINGO

INT-CL (IPC): C08 L 9/06; B60 C 1/00; C08 K 3/36; C08 K 5/54; C08 L 9/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWIC

28. Document ID: JP 09111001 A

L30: Entry 28 of 36

File: JPAB

Apr 28, 1997

PUB-NO: JP409111001A
DOCUMENT-IDENTIFIER: JP 09111001 A
TITLE: PRODUCTION OF CARBON BLACK/RESIN MASTERBATCH

PUBN-DATE: April 28, 1997

INVENTOR-INFORMATION:

NAME

COUNTRY

BULL, JR CHARLES L

COLVIN, HOWARD A

INT-CL (IPC): C08 J 3/22

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWIC

29. Document ID: JP 07309977 A

L30: Entry 29 of 36

File: JPAB

Nov 28, 1995

PUB-NO: JP407309977A
DOCUMENT-IDENTIFIER: JP 07309977 A
TITLE: PRODUCTION OF RUBBER VULCANIZATE

PUBN-DATE: November 28, 1995

INVENTOR-INFORMATION:

NAME

COUNTRY

COLVIN, HOWARD A

BULL, JR CHARLES L

q

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Printer: cp3_4c07_gbgmptr

Date: 09/20/02

Time: 15:16:26

Document Listing

Document	Selected Pages	Page Range
US006040009	39	1 - 39
US005393823	8	1 - 8
US004972027	22	1 - 22
US004923945	16	1 - 16
Total (4)	85	-

INT-CL (IPC): C08 L 21/00; C08 K 7/16

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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RWD

30. Document ID: JP 05214252 A

L30: Entry 30 of 36

File: JPAB

Aug 24, 1993

PUB-NO: JP405214252A
DOCUMENT-IDENTIFIER: JP 05214252 A
TITLE: COMPATIBILIZED POLYMERIC SYSTEM

PUBN-DATE: August 24, 1993

INVENTOR-INFORMATION:

NAME

COUNTRY

COLVIN, HOWARD A

ALSAMARRAIE, MUHANAD A

PARKER, DANE KENTON

INT-CL (IPC): C08L 101/00; C08G 18/40; C08G 81/02

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

RWD

31. Document ID: JP 05214167 A

L30: Entry 31 of 36

File: JPAB

Aug 24, 1993

PUB-NO: JP405214167A
DOCUMENT-IDENTIFIER: JP 05214167 A
TITLE: POLYMER DERIVED FROM CONJUGATED DIOLEFIN, VINYL-SUBSTITUTED AROMATIC COMPOUND
AND OLEFINICALLY UNSATURATED NITRILE

PUBN-DATE: August 24, 1993

INVENTOR-INFORMATION:

NAME

COUNTRY

SENYEK, MICHAEL L

COLVIN, HOWARD A

INT-CL (IPC): C08L 9/00; C08L 9/00; C08F 236/04; C08L 7/00; C08L 23/16; C08L 25/10

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

RWD

32. Document ID: JP 03106831 A

L30: Entry 32 of 36

File: JPAB

May 7, 1991

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UserID: q

Printer: cp3_4c07_gbgnptr

Summary

Document	Pages	Printed	Missed
US006171381	16	16	0
US006171381	16	16	0
Total (2)	32	32	0

PUB-NO: JP403106831A
DOCUMENT-IDENTIFIER: JP 03106831 A
TITLE: IMPROVED THERMAL DIMERIZATION OF ISOPRENE

PUBN-DATE: May 7, 1991

INVENTOR-INFORMATION:

NAME

COUNTRY

COLVIN, HOWARD A

INT-CL (IPC): C07C 13/20; B01J 31/02; C07C 2/42; C07C 13/263; C07B 61/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWIC

33. Document ID: JP 03020323 A

L30: Entry 33 of 36

File: JPAB

Jan 29, 1991

PUB-NO: JP403020323A
DOCUMENT-IDENTIFIER: JP 03020323 A
TITLE: CROSSLINKABLE RUBBER COMPOSITION

PUBN-DATE: January 29, 1991

INVENTOR-INFORMATION:

NAME

COUNTRY

WEINSTEIN, ARTHUR H

COLVIN, HOWARD A

PARKER, DANE KENTON

INT-CL (IPC): C08G 18/81; C08L 15/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWIC

34. Document ID: JP 02075640 A

L30: Entry 34 of 36

File: JPAB

Mar 15, 1990

PUB-NO: JP402075640A
DOCUMENT-IDENTIFIER: JP 02075640 A
TITLE: RUBBER VALCANIZING AGENT

PUBN-DATE: March 15, 1990

INVENTOR-INFORMATION:

NAME

COUNTRY

COLVIN, HOWARD A

BULL, JR CHARLES L

MAGNUS, FREDRICK LEWIS

q

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Date: 09/19/02

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Document Listing

Document	Selected Pages	Page Range
US006171381	16	1 - 16
US006171381	16	1 - 16
Total (2)	32	-

INT-CL (IPC): C08L 21/00; C07C 321/00; C08K 5/36; C08K 11/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWOC
Draw Desc	Image									

35. Document ID: JP 01278515 A

L30: Entry 35 of 36

File: JPAB

Nov 8, 1989

PUB-NO: JP401278515A

DOCUMENT-IDENTIFIER: JP 01278515 A

TITLE: DIAMINE-BASED CURING AGENT FOR AWES-NEGATIVE POLYURETHANE

PUBN-DATE: November 8, 1989

INVENTOR-INFORMATION:

NAME

COUNTRY

COLVIN, HOWARD A

INT-CL (IPC): C08G 18/32; C08G 18/66

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWOC
Draw Desc	Image									

36. Document ID: JP 01204938 A

L30: Entry 36 of 36

File: JPAB

Aug 17, 1989

PUB-NO: JP401204938A

DOCUMENT-IDENTIFIER: JP 01204938 A

TITLE: POLYMER ALLOY

PUBN-DATE: August 17, 1989

INVENTOR-INFORMATION:

NAME

COUNTRY

MUSE, JR JOEL

COLVIN, HOWARD A

INT-CL (IPC): C08L 21/00; C08L 9/02; C08L 23/26; C08L 23/26; C08L 101/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWOC
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COATED STRUCTURAL ARTICLES

This is a continuation-in-part of application Ser. No. 08-885,760 filed Jun. 27, 1997 now abandoned.

BACKGROUND OF THE INVENTION

This invention relates to structural articles and a method for making such articles comprising a substrate having an ionic charge coated with a coating having essentially the same charge and consisting essentially of a filler material and a binder material.

For many years substrates such as fiberglass have been coated with various compositions to produce structural articles having utility in, among other applications, the building industry. U.S. Pat. No. 5,001,005 relates to structural laminates made with facing sheets. The laminates described in that patent include thermosetting plastic foam and have planar facing sheets comprising 60% to 90% by weight glass fibers (exclusive of glass micro-fibers), 10% to 40% by weight non-glass filler material and 1% to 30% by weight non-asphaltic binder material. The filler materials are indicated as being clay, mica, talc, limestone (calcium carbonate), gypsum (calcium sulfate), aluminum trihydrate (ATH), antimony oxide, cellulose fibers, plastic polymer fibers or a combination of any two or more of those substances. The patent further notes that the filler materials are bonded to the glass fibers using binders such as urea-, phenol- or melamine-formaldehyde resins (UF, PF, and MF resins), or a modified acrylic or polyester resin. Ordinary polymer latexes used according to the disclosure are Styrene-Butadiene-Rubber (SBR), Ethylene-Vinyl-Chloride (EVC), PolyVinylidene Chloride (PvdC), modified PolyVinyl Chloride (PVC), PolyVinyl Alcohol (PVOH), and Poly-Vinyl Acetate (PVA).

U.S. Pat. No. 4,745,032 discloses an acrylic coating comprised of one acrylic underlying resin which includes fly ash and an overlying acrylic resin which differs from the underlying resin.

U.S. Pat. No. 4,229,329 discloses a fire retardant coating composition comprising fly ash and vinyl acrylic polymer emulsion. The fly ash is 24 to 50% of the composition.

Many different coating compositions have been formulated over the years but often such compositions would bleed through substrates, such as fiberglass substrates, if the substrates were coated on just one side, unless the compositions had a high binder content and/or included viscosity modifiers to enhance the viscosity of the coating composition. To prevent bleed through, such coating compositions sometimes had their viscosity increased by blowing or whipping air into the compositions. Although such blown compositions did not bleed through to the other side of mats such as fiberglass mats, the raw material costs for the compositions were high because of the numbers of constituent elements involved.

Accordingly, it is an object of this invention to provide a structural article having a coating which includes only two major constituents, while eliminating the need for viscosity modifiers, for stabilizers or for blowing. It is also an object of this invention to provide a low cost, relatively light weight structural article comprised principally of a coating having a low binder content and a high filler content. It is a further object of this invention to provide a relatively light weight, low cost coating which coats a substrate without bleeding through the substrate.

SUMMARY OF THE INVENTION

In accordance with the invention, a structural article is made by coating a substrate having an ionic charge with a

coating having essentially the same ionic charge. The coating consists essentially of a filler material and a binder material. By coating the substrate with a coating having essentially the same ionic charge, the applicant has developed a zero bleed through product while using only two major ingredients in the coating and eliminating the need for costly and time consuming processing steps such as blowing. Applicant has discovered that by producing a coating having essentially the same ionic charge as the substrate, a zero bleed through product may be produced having a low binder content and no viscosity modifiers.

The coated substrate of the present invention may be any suitable reinforcement material capable of withstanding processing temperatures, such as glass fibers, polyester fibers, cellulosic fibers, asbestos, steel fibers, alumina fibers, ceramic fibers, nylon fibers, graphite fibers, wool fibers, boron fibers, carbon fibers, jute fibers, polyolefin fibers, polystyrene fibers, acrylic fibers, phenolformaldehyde resin fibers, aromatic and aliphatic polyamide fibers, polyacrylamide fibers, polyacrylimide fibers or mixtures thereof which may include bicomponent fibers.

The filler may be class F fly ash wherein 90% to 95% by weight of the fly ash is aluminosilicate. Such a fly ash, known as Alsil O4TR, is produced by JTM Industries of Kennesaw, Ga. Alternatively, the filler may be charged calcium carbonate or ceramic microspheres, or a blend of fly ash and calcium carbonate, or a blend of fly ash, calcium carbonate and ceramic microspheres or any combination of these filler materials to meet desired cost and weight criteria. Calcium carbonate and fly ash filler increase the weight of the product, but utilization of ceramic microspheres enables the manufacture of a product with reduced weight and increased fire resistant properties. Ceramic microspheres can withstand heat greater than 2000° F. Also, ceramic microspheres increase compressive strength, absorb no latex and/or water and thus permit the faster drying of the product. Ceramic microspheres also increase product flexibility.

Further, the ceramic microspheres help to increase the pot life of the coating. Heavier particles in the calcium carbonate and fly ash filler, although they may comprise but a small percentage of the particles in the filler, have a tendency to settle near the bottom of a storage vessel. When ceramic microspheres are mixed together with calcium carbonate and/or fly ash filler, a dispersion is produced which has an increased pot life or shelf life. Without wishing to be bound by any particular theory, it is believed that as the filler particles naturally fall in the vessel and the ceramic microspheres rise, the smaller size filler particles are supported by the ceramic microspheres, thus enabling the microspheres to stay in solution and preventing the filler particles, to at least some extent, from descending to the bottom of the vessel.

The table below provides, in percentages, some of the combinations of calcium carbonate, fly ash and ceramic microspheres which applicant has utilized as the filler component in the coating:

TABLE I

	A %	B %	C %	D %	E %	F %
1. Water	18.9	25.9	37.33	25.9	24.9	24.9
2. Acrylic Latex	6.0	6.0	6.42	6.0	6.0	6.0
3. Fly Ash	75.0	34.0	—	40.0	—	20.0
4. CaCO ₃	—	34.0	—	—	40.0	20.0
5. Microspheres	—	—	56.14	28.0	29.0	29.0
6. Defoamer	0.1	0.1	0.1	0.1	0.1	0.1
	100%	100%	100%	100%	100%	100%

The microspheres were a 50/50 ratio of 3M's W1012 microspheres and 3M's smaller diameter G200 micro-

Term	Documents
COLVIN.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	3063
COLVINS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1
HOWARD.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	84840
HOWARDS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	113
\$	0
A[DWPI,EPAB,JPAB,USPT,PGPB]	21347867
AA[DWPI,EPAB,JPAB,USPT,PGPB]	65495
AAA[DWPI,EPAB,JPAB,USPT,PGPB]	14044
AAAA[DWPI,EPAB,JPAB,USPT,PGPB]	1752
AAAAA[DWPI,EPAB,JPAB,USPT,PGPB]	751
AAAAAA[DWPI,EPAB,JPAB,USPT,PGPB]	575
(COLVIN, HOWARD \$ [IN]).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	36

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spheres. Although the table shows possible combinations of calcium carbonate, fly ash and ceramic microspheres in the filler component of the coating, it is believed that any combination of these materials may be employed.

The coating is prepared by using a binder material such as a high performance heat-reactive acrylic latex polymer to bond the filler materials together and to bond the filler to the substrate. Such a binder material is Hycar 2679 acrylic latex polymer supplied by B.F. Goodrich Company of Cleveland, Ohio. It is believed, however, that any linear polymer, linear copolymer or branched polymer may be useful in preparing the coating. Possible binder materials include butyl rubber latex, SBR latex, neoprene latex, polyvinyl alcohol emulsion, SBS latex, water based polyurethane emulsions and elastomers, vinyl chloride copolymers, nitrile rubbers and polyvinyl acetate copolymers.

In a preferred embodiment (Examples III, V and VIII below), the coating comprises nearly 85% by weight of the structural article. In that coating, approximately from 84% to 96% by weight is filler and the remainder is the acrylic latex binder. The filler is approximately 50% fly ash and 50% calcium carbonate. The substrate comprises about 15% by weight of the structural article. Glass fibers comprise approximately 12% by weight of the article and a binder material comprises about 3% by weight of the article. The binder which bonds together the glass fibers is from 99% to 75% (preferably 98% to 94%) by weight urea formaldehyde and from 1% to 25% (preferably 2% to 6%) by weight standard acrylic latex.

The substrate may be coated by air spraying, dip coating, knife coating, roll coating or film application such as lamination/heat pressing. The coating may be bonded to the substrate by chemical bonding, mechanical bonding and/or thermal bonding. Mechanical bonding is achieved by force feeding the coating onto the substrate with a knife.

Structural articles made in accordance with this invention may be of any shape and may be used in any of a variety of products including roofing shingles, structural laminate facing sheets, building air duct liners, roofing underlayment (or roofing felt), underlayment for organic shingles to provide Class "A" U.L. rating, built up roofing materials, roll roofing, modified roll products, filter media (including automotive filters), automotive hood liners, head liners, fire walls, vapor barriers etc. Preferably, such articles are planar in shape. The substrate is coated on one side or both sides depending on the intended application. For instance, if one side of the substrate is coated with the filler/binder coating, the other surface can be coated with conventional roofing asphalt, modified asphalts and non-asphaltic coatings, and the article can then be topped with roofing granules. It is believed that such roofing material could be lighter in weight, offer better fire resistance and better performance characteristics (such as cold weather flexibility, dimensional stability and strength) than prior art roofing materials.

Additionally, the structural article may be coated with a water repellent material. Two such water repellent materials are Aurapel 330R and Aurapel 391 available from the Auralux Corporation of Norwich, Conn. It is believed that wax emulsions, oil emulsions, silicone emulsions, polyolefin emulsions and surfonys as well as other similar performing products may also be suitable water repellent materials. Further, structural articles made in accordance with the invention may be coated with an algicide such as zinc powder, copper oxide powder or the herbicides Atrazine available from e.g. Ribelin Industries or Diuron available from e.g. Olin Corporation, an antifungal material such as Micro-Chek 11P, an antibacterial material such as Micro-Chek 11-S-160, a surface friction agent such as Byk-375, a flame retardant material such as ATH (aluminum trihydrate) available from e.g. Akzo Chemicals and antimony oxide

available from e.g. Laurel Industries and/or a coloring dye such as T-1133A and iron oxide red pigments, and other products which can impart specific surface functions. The Micro-Chek products are available from the Ferro Corporation of Walton Hills, Ohio. Byk-375 may be obtained from Wacker Silicone Corporation of Adrian, Mich. and T-1133A is sold by Abco Enterprises Inc. of Allegan, Mich. The additional coatings of, e.g. water repellent material, antifungal material, antibacterial material, etc., may be applied to one or both sides of structural articles otherwise having filler/binder coatings on one or both sides of a substrate. For example, structural articles comprising substrates coated on one or both sides with filler/binder coatings could be coated on one side with a water repellent composition and on the other side with an antibacterial agent.

Applicant's invention also involves a method for making a structural article comprising the steps of coating a substrate having an ionic charge with a coating having essentially the same ionic charge. The coating consists essentially of a filler material and a binder material. In one embodiment, the coating is prepared by mixing the filler material and the binder material until the ionic charge of the mixed materials changes such as to increase the viscosity of the coating. In the preferred embodiment, the substrate is anionic and the coating is essentially anionic even though the cationic nature of the coating increases during the aforementioned mixing.

DETAILED DESCRIPTION

Structural articles are made by coating a substrate having an anionic charge with a coating having essentially the same ionic charge. Any suitable reinforcement material capable of withstanding processing temperatures may be employed as a substrate in accordance with the invention. Examples include, inter alia, glass, fiberglass, ceramics, graphite (carbon), PBI (polybenzimidazole), PITE, polyaramides, such as KEVLAR and NOMEX, metals including metal wire or mesh, polyolefins such as TYVEK, polyesters such as DACRON or REEMAY, polyamides, polyimides, thermoplastics such as KYNAR and TEFZEL, polyether sulfones, polyether imide, polyether ketones, novoloid phenolic fibers such as KYNOL, cotton, asbestos and other natural as well as synthetic fibers. The substrate may comprise a yarn, filament, monofilament or other fibrous material either as such or assembled as a textile, or any woven, non-woven, knitted, matted, felted, etc. material. The polyolefin may be polyvinyl alcohol, polypropylene, polyethylene, polyvinyl chloride, polyurethane, etc. alone or in combination with one another. The acrylics may be DYNEL, ACRYLAN and/or ORLON. RHOPLEX AC-22 and RHOPLEX AC-507 are acrylic resins sold by Rohm and Haas which may also be used. The cellulose fibers may be natural cellulose such as wood pulp, newsprint, Kraft pulp and cotton and/or chemically processed cellulose such as rayon and/or lyocell.

The fly ash referred to in the examples was obtained from JTM Industries, Inc. of Martin Lake and Jewett, Tex. and had a particle size such that less than 0.03% remained on an agitated 0.1 inch x 0.1 inch screen. The ceramic microspheres were manufactured by Zeelan Industries of 3M Center Bldg., 220-8E-04, St. Paul, Minn. 55144-1000. Calcium carbonate was obtained from Franklin Industrial Minerals of 612 Tenth Avenue North, Nashville, Tenn. 37203. Black colorant or pigment used in various of the articles of the examples was T-113A sold by Abco, Inc.

Foamed structural articles made in accordance with the present invention may be made by any of the known methods for making foamed compositions such as, for example, aeration by mechanical mixing and the other techniques described in U.S. Pat. No. 5,110,839.

EXAMPLE I

To reduce the weight and cost of coated structural articles, the applicant formulated the coating using three ingredients:

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Chen

Search Results - Record(s) 1 through 10 of 10 returned.**1. Document ID: US 20020128370 A1**

L31: Entry 1 of 10

File: PGPB

Sep 12, 2002

PGPUB-DOCUMENT-NUMBER: 20020128370

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020128370 A1

TITLE: Reinforced silica/elastomer composite

PUBLICATION-DATE: September 12, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Colvin, Howard Allen	Arlington	TX	US	
<u>Chen, Sun Lin</u>	Akron	OH	US	

US-CL-CURRENT: 524/493

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

2. Document ID: US 5516818 A

L31: Entry 2 of 10

File: USPT

May 14, 1996

US-PAT-NO: 5516818

DOCUMENT-IDENTIFIER: US 5516818 A

TITLE: Method for removing small amounts of high boiling point organic compound from aqueous polymerization products

DATE-ISSUED: May 14, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
<u>Chen; Sun-Lin</u>	Akron	OH		
Wong; Tang H.	Hudson	OH		
Gujarathi; Ramesh N.	Munroe Falls	OH		

US-CL-CURRENT: 523/332, 523/328, 523/330, 523/340, 528/490, 528/491, 528/493, 528/496, 528/498, 528/499, 528/500, 528/502R, 528/934

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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water, BF Goodrich acrylic latex Hycar 2679 and JTM Alsil O4TR fly ash filler. The amounts of the three constituents were as follows: 19% water, 6% Hycar 2679, 74% JTM fly ash. Dye and defoaming agent made up 1% of the formulation. Generally, the coating may be produced by combining 50% to 80% fly ash filler, 1% to 25% acrylic latex binder, 15% to 25% water and minor amounts of dye and defoaming agent as needed. The defoaming agent was Drew Plus Y-250 sold by Drews Industrial Division of Boonton, N.J. The materials were mixed in a reaction or mixing kettle for 45 minutes. JTM fly ash filler comprised a much higher percentage of the coating than the 40-45% by weight filler which is the industry standard. Moreover, the binder content was lower than amounts usually found in such coating formulations.

The coating was used to coat a fiberglass mat on one side. The mat was manufactured by Elk Corporation of Ennis, Tex. and had a basis weight in the range of 1.4 lb./sq. to 2.0 lb./sq. The mat had a porosity in the range of 800 to 1,000 cfm. Heretofore, when such highly porous mats have been coated on one side only, it was expected that the coating would bleed through to the other side. In accordance with the present invention however, the novel coating coated the surface of the fiberglass mat very well and did not bleed through to the other side of the mat. The coated article was durable and flexible and did not crack on bending. Typical tensile strength measurements were as follows: machine direction 156 lbs.; cross direction 57 lbs.; average 107 lbs. Typical tear strength measurements were as follows: machine direction 151 grams; cross direction 306 grams; average 229 grams.

The coated article was hand brushed with adhesive to determine if there was bleed through to the other side of the article. No bleed through was observed. Further, the coated article was soaked in unleaded gasoline for 48 hours and no change in the physical state of the article was observed. There was no degradation and no reaction. The coated article was also checked for combustibility. When exposed to flame it burned, but when the flame was removed the burning stopped.

Surprisingly, when the coating of the present invention was used to coat the fiberglass mat on one side, it did not bleed through to the other side even though the coating had a relatively low viscosity of 700 cp. Although not wishing to be bound by any particular theory, the applicant believes that the coating did not bleed through the mat because the fiberglass mat is anionic and the coating of the present invention (when wet) includes a combination of water and Hycar 2679 (which together are anionic) and JTM filler (which is also anionic). The addition of the JTM filler to the acrylic latex in water results in a repulsion of charges and low viscosity. Although low viscosity is not a desired objective for coating a highly porous mat, the unique characteristic of the invention is that the coating does not bleed through regardless of the viscosity because the mat is also anionic and like charges repel each other just as the north pole of one magnet repels the north pole of another magnetic.

If desired, however, the viscosity of the coating can be increased through mixing. It is believed that JTM Alsil-O4TR fly ash filler is approximately 90% to 95% aluminosilicate, which is unaffected by water, but in acidic solution undergoes hydrolysis. The water and latex solution to which JTM fly ash filler was added is acidic in nature and, on prolonged mixing, there is some hydrolyzation of the aluminosilicate thereby increasing the viscosity of the coating. The longer or the more rapidly the coating is mixed, the higher the viscosity. However, the coating still maintains an essentially anionic charge and thus there is still repelling of charges between the coating and the substrate.

Whether slowly or rapidly mixed, the coatings of the present invention may be applied to the substrates in relatively uniform thin coats because the like charges among the filler and acrylic latex elements in the coating repel one another. Thus, it is believed that the ionic charge repulsion characteristic which prevents the coating from bleeding through the mat also enables the application on the mat of a relatively uniform thin film coating. In instances where, due to price, supply or other considerations, the filler material to be employed has an ionic charge which is essentially the opposite of the charge of the substrate, modifiers are available to coat the filler material so that ultimately the coating and substrate of the article have essentially the same ionic charge. It is believed that viscosity modifiers could serve such a purpose.

EXAMPLE II

Water, Hycar 2679 acrylic latex and JTM Alsil-O4TR fly ash were combined in the same amounts as noted in Example I. However, in accordance with another embodiment of the invention, the materials were mixed for a longer period, 7 hours, in the same reaction/mixing vessel with a paddle mixer. The coating had a viscosity of about 40,000 cp. The coating was then applied to the same type of mat using the same technique as recited in Example I. The tensile and tear strengths of the coated article were the same as in Example I. The viscosity of the materials increased during mixing in both Examples I and II. However, relatively rapid mixing such as in Example I results in the production of foam which was observed in only small amounts in Example II which was mixed more slowly. Accordingly, a defoaming agent is added when the materials are mixed rapidly.

The invention provides a coated fabric which is rigid in nature and is also flexible enough to be rolled up, showing no signs of cracking, etc. The coated fabric has a porosity of less than 1.0 cfm and adheres very well to polyurethane foam, isocyanurate foam, asphaltic compounds, and granules (non-asphaltic shingle components).

The coated product may have few pinholes or may have numerous pinholes and still maintain a porosity of less than 1.0 cfm when coated with solvent based adhesive such as Firestone Bonding Adhesive BA-2004, i.e. the adhesive did not bleed to other side.

The coated articles were made water repellent by coating with further additives, Aurapel 330R and Aurapel 391, which can be obtained from Auralux Corporation. The coating was accomplished by diluting the coating compound with water and then kiss coating the articles on one side while they were being coated on the other side by standard coating techniques which included the use of a doctor blade.

Prior to coating with a water repellent coating, the novel coating of the present invention can be treated with pigment or dye or any other suitable coloring means to give color to the structural articles of the invention. For instance, a carbon black pigment (0.5% by weight) was added to the coating composition of the Examples to give color texture to the finished coating on the fiberglass mat.

Besides water repellent treatment, the structural articles of the present invention can be coated with antifungal, antibacterial and surface friction agents, an algacide and/or a flame retardant material by mixing with the coating constituents prior to coating the substrate or by spraying on the partly finished articles at some point in the processing, e.g. between drying and curing.

Coating of the fiberglass substrates was accomplished by the applicant using a hand-held coater which can be obtained from the Gardner Company, but any conventional method, such as spraying, dipping and flow coating from aqueous or solvent dispersion, calendering, laminating and the like,

3. Document ID: US 5284905 A

L31: Entry 3 of 10

File: USPT

Feb 8, 1994

US-PAT-NO: 5284905

DOCUMENT-IDENTIFIER: US 5284905 A

TITLE: Process for producing stable latex

DATE-ISSUED: February 8, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Chen; Sun-Lin	Akron	OH		
Gujarathi; Ramesh N.	Akron	OH		
Prentice; Susan M.	Akron	OH		

US-CL-CURRENT: 524/710; 523/201, 523/319, 523/322, 524/457, 524/458, 524/460

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

4. Document ID: US 5191009 A

L31: Entry 4 of 10

File: USPT

Mar 2, 1993

US-PAT-NO: 5191009

DOCUMENT-IDENTIFIER: US 5191009 A

TITLE: Process for producing stable latex

DATE-ISSUED: March 2, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Chen; Sun-Lin	Akron	OH		
Gujarathi; Ramesh N.	Akron	OH		
Prentice; Susan M.	Akron	OH		

US-CL-CURRENT: 524/460; 524/458, 524/710, 524/833

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

5. Document ID: US 5171768 A

L31: Entry 5 of 10

File: USPT

Dec 15, 1992

US-PAT-NO: 5171768

DOCUMENT-IDENTIFIER: US 5171768 A

TITLE: Process for the production of carboxylated latexes by the selective monomer addition and polymerization

DATE-ISSUED: December 15, 1992

followed by drying and baking, may be employed to coat the substrate as is well known in the art. Best coating results were observed using a Gardner profile 10 blade. After coating, the samples were placed in an oven at approximately 400° for about 2.0 minutes to achieve drying and curing. Additionally, the coating may be separately formed as a film of one or more layers for subsequent combination with the substrate.

Hycar 2679 acrylic latex polymer has a low Brookfield viscosity of 100 cP. The low viscosity makes the polymer easily miscible with water and filler. This heat reactive acrylic polymer is compatible with all fillers due to its anionic charge. Products made with coatings which include the polymer are flexible at extreme high and low temperatures because the polymer has a glass transition temperature (T_g) of -3° C.

Hycar 2679 polymer emulsion contains a colloidal dispersion of polymer and copolymers in water, emulsifiers, such as synthetic soap (sometimes known as surface active agents or surfactants) and other ingredients such as buffers and protective colloids. These ingredients enable the polymer to be compatible with a wide variety of fillers. Without being bound to any particular theory, it is believed that Hycar 2679, with its thixotropic characteristics, enhances the viscosity of most fillers.

In order to increase the pot life and lower the viscosity of the coating compositions of Examples I and II and to decrease the basis weight and increase the coefficient of friction of the finished coated product, calcium carbonate was incorporated into the formulation in a ratio of 50:50 and 25:75 to JTM fly ash filler. Calcium carbonate is produced by crushing limestone. During this crushing process, trace amounts of iron and aluminum which come off of the grinding apparatus provide an ionic charge to the calcium carbonate, permitting it to substitute for some or all of the fly ash in the coating. The calcium carbonate can be 100% of the filler component if an appropriate ionic charge is present or if an appropriate ionic material is added (either instance being referenced herein as "charged calcium carbonate"). Suitable ionic materials include Alcosperse 149 which may be obtained from Alco Chemical Division of National Starch and Chemicals of Chattanooga, Tenn., Aerosol 18 which may be obtained from American Cyanamid in West Patterson, N.J., ammonium stearate and ammonium hydroxide, both of which may be obtained from Henkel of Ambler, Pa. Calcium carbonate is more stable in water when compared to JTM fly ash and does not undergo hydrolysis in acidic media created by the Hycar acrylic latex and the water. Calcium carbonate has a larger particle size than JTM fly ash. The mixture of small and large size particles makes the surface of the coated product rough and increases the coefficient of friction.

EXAMPLE III

When the filler component in the coating was 50% JTM fly ash and 50% calcium carbonate, the coating was very fluid (viscosity=272 cp. to 388 cp) and had a pot life of 6 to 8 hours. The coated product had a porosity of 3.5 cfm.

EXAMPLE IV

A coating formulation was prepared wherein the filler component comprised 25% calcium carbonate and 75% JTM fly ash. First, 221.59 lbs. of calcium carbonate were added to 342.81 lbs. of water forming a solution. The Hycar latex was added and 4 inches to 6 inches of foam were generated. 664.76 lbs. of fly ash were then added and most of the foam was eliminated. 1.3 lbs. of defoamer were then added and the remaining foam was eliminated. The coating was mixed for about 15 minutes until a thixotropic effect

was observed. The coating was very fluid (viscosity=2,000 cp) and compound density was about 13 lbs. gallon. The fiberglass mat described in Examples I and II was coated using a blade and no bleed through was observed.

EXAMPLE V

The fiberglass mat described in Examples I and II was coated on one side with a coating according to Example I except that 50% of the JTM fly ash was replaced with calcium carbonate. Two rolls of fiberglass mat were coated on one side and, due to the ionic nature of the coating, no bleed through of the coating was noticed on the other side of the mat. The low viscosity coating composition coated the fiberglass mat fabric as well as the higher viscosity composition. Utilization of a low viscosity coating enabled a reduction in basis weight of the final product from 10.5 lbs./sq. to 8.5 lbs./sq.

EXAMPLE VI

The fiberglass mat of Examples I and II was coated on both sides using the coating formulation of Example IV. The top of the fabric was coated using a straight edge blade while the bottom of the fabric was kiss coated using a large steel roller which picked up the coating from a tank and coated the bottom of the fabric. The coating in the tank had been diluted by increasing the water composition 10% to lower the viscosity to facilitate kiss coating.

EXAMPLE VII

Ceramic microspheres were incorporated in the filler component of the coating together with JTM fly ash and calcium carbonate to produce one sided and two sided coated products as indicated in Table I above. Microspheres offer a variety of inherent advantages over filler materials having irregularly shaped particles such as JTM fly ash and calcium carbonate. Advantages include improved flow, low viscosity even when the coating has a high filler content, hardness and abrasion resistance and low dielectric constant. Utilization of ceramic microspheres enhances one of the most important physical properties of the finished coated product—thermal insulation. Accordingly, use of ceramic microspheres in the coating of the product will enable facer manufacturers to use less foam while achieving the same Class A fire rating. Ceramic microspheres also bond ionically to water and the Hycar 2679 acrylic latex and require no additive to build up viscosity.

EXAMPLE VIII

It is believed that a preferred embodiment is prepared by combining constituents in the following amounts: 29% water, 6% Hycar 2679 acrylic latex, 31.25% JTM fly ash, 31.25% calcium carbonate, 0.5% Aurapel 330R and 2.0% SBR rubber which was obtained from Ultra Pave, a distributor of Goodyear Tire and Rubber Co. of Cleveland, Ohio. Alternative suitable SBR rubbers include UP1156, UP70 and UP72, all of which may be obtained from Ultra Pave. A cross linking agent, such as B720 obtained from Texpar of Waukesha, Wis. may be added to cross link the acrylic latex and the SBR rubber. Zinc oxide may also be employed as a cross linking agent.

The inventive coatings described in the foregoing examples did not bleed through to the other side of the fiberglass mats even when the viscosity of the coating composition was very low and the fiberglass mat utilized was highly porous. JTM fly ash contains aluminum, silicon, calcium, magnesium and silicon dioxide. All of these ingredients are cationic in nature and show a great affinity toward the anionic charge of the Hycar acrylic latex polymer. This

INVENTOR-INFORMATION:

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US-CL-CURRENT: 523/348; 523/353, 524/458, 524/460, 524/822

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw Desc	Image									

6. Document ID: US 4609434 A

L31: Entry 6 of 10

File: USPT

Sep 2, 1986

US-PAT-NO: 4609434

DOCUMENT-IDENTIFIER: US 4609434 A

**** See image for Certificate of Correction ****

TITLE: Composite sheet prepared with stable latexes containing phosphorus surface groups

DATE-ISSUED: September 2, 1986

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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<u>Chen; Sun-Lin</u>	Midland	MI		

US-CL-CURRENT: 162/168.1; 162/169

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw Desc	Image									

7. Document ID: US 4506057 A

L31: Entry 7 of 10

File: USPT

Mar 19, 1985

US-PAT-NO: 4506057

DOCUMENT-IDENTIFIER: US 4506057 A

TITLE: Stable latexes containing phosphorus surface groups

DATE-ISSUED: March 19, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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<u>Chen; Sun-Lin</u>	Midland	MI		

US-CL-CURRENT: 524/461; 524/193, 524/710

affinity of opposite charges results in high viscosity of the coating compound. Fly ash is inert in water, but it hydrolyzes in acidic media. Hycar 2679 latex is acidic in nature, having a pH of 3.7. This latex hydrolyzes the JTM fly ash and this hydrolysis results in ever increasing viscosity of the composition over time. JTM fly ash also absorbs water over time and this water absorption further increases the composition's viscosity. After about 3 hours, the viscosity of the composition has been raised to such an extent that the addition of water is required to properly adjust it.

To increase the pot life of the coating composition, JTM fly ash was replaced in part with calcium carbonate filler. The calcium carbonate not only reduced the composition's viscosity, it also lowered the basis weight of the finished coated product. Further, while calcium carbonate has some affinity toward the Hycar acrylic latex, it has much less affinity than JTM fly ash. The aluminum and iron impurities in calcium carbonate, which result from the crushing of limestone by metal rods, are cationic in nature and cause the affinity of the calcium carbonate for the Hycar latex. However, the metal impurities in the calcium carbonate weigh far less than JTM filler particles. Utilization of calcium carbonate filler blends as 100% of the filler component or some portion thereof in conjunction with JTM fly ash prolongs the pot life of the coating composition. JTM fly ash particles are more uniform and smaller in size when compared to calcium carbonate filler particles. Accordingly, products having a coating composition which includes only JTM fly ash as filler, have a smoother surface than products which are coated with a composition which includes calcium carbonate. Moreover, products including calcium carbonate as a filler component in the coating have a reduced final basis weight because fewer of the larger calcium carbonate particles are required to block the porous fiberglass mat.

Utilization of ceramic microspheres as a filler component in the coating also enhances the pot life of the coating compound. Ceramic microspheres do not absorb water, but bond with water and the Hycar acrylic latex with weak hydrogen bonding and this bonding phenomena results in a thixotropic effect which results in the desired viscosity for long pot life, composition stability, improved flow and ease of mixing.

It should be understood that the above examples are illustrative, and that compositions other than those described above can be used while utilizing the principals underlying the present invention. For example, other sources of filler as well as mixtures of acrylic latex and/or surfactants can be used in formulating the structural articles. Moreover, the coating compositions can be applied to various types of substrates, as described above.

What is claimed is:

1. A structural article comprising a substrate having an ionic charge coated with a coating having essentially the same ionic charge wherein said coating consists essentially of a filler material and a binder material and wherein said binder material bonds the filler material together and to the substrate and wherein said coatings does not bleed through said substrate.

2. A structural article according to claim 1 wherein said substrate is fiberglass, said filler is selected from the group consisting of fly ash, calcium carbonate, ceramic microspheres and mixtures thereof and said binder is acrylic latex.

3. A structural article according to claim 2 wherein said substrate is planar and is coated on one side with said coating.

4. A structural article according to claim 2 wherein said substrate is planar and is coated on both sides with said coating.

5. A structural article according to claims 1, 3 or 4 wherein said article further includes on one or both sides a water repellent material.

6. A structural article according to claims 1, 3 or 4 wherein said article further includes on one or both sides an anti-fungal material.

7. A structural article according to claims 1, 3 or 4 wherein said article further includes on one or both sides an antibacterial material.

8. A structural article according to claims 1, 3 or 4 wherein said article further includes on one or both sides a surface friction agent.

9. A structural article according to claims 1, 3 or 4 wherein said article further includes on one or both sides a flame retardant material.

10. A structural article according to claims 1, 3 or 4 wherein said article further includes on one or both sides an algicide.

11. A structural article according to claims 1, 3 or 4 wherein said article is colored with dye on one or both sides.

12. A structural article according to claims 2, 3 or 4 wherein said substrate is bonded together by a binder material consisting essentially of urea formaldehyde and acrylic latex.

13. A structural article coated with a coating consisting essentially of a filler material and a binder material wherein

a) said article is from 10% to 25% by weight glass fibers and

b) said coating is from 84% to 96% filler selected from the group consisting of fly ash, charged calcium carbonate, ceramic microspheres and mixtures thereof and from 16% to 4% acrylic latex binder material.

14. A structural article according to claim 13 wherein said coating further includes SBR rubber.

15. A structural article according to claim 14 wherein said acrylic latex binder and said rubber are cross linked.

16. A structural article according to claim 15 wherein said glass fibers are bonded together by a mixture of from 99% to 75% urea formaldehyde and from 1% to 25% acrylic latex.

17. A method for making a structural article comprising the steps of coating a substrate having an ionic charge with a coating having essentially the same ionic charge wherein said coating consists essentially of a filler material and a binder material and wherein said binder material bonds the filler material together and to the substrate.

18. A method according to claim 16 wherein

a) said coating is prepared by mixing the filler material and the binder material until the viscosity of the coating increases; and

b) the substrate is then coated with the more viscous coating.

19. A structural article according to claim 14 wherein said article is roofing underlayment and wherein said filler is charged calcium carbonate.

* * * * *

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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8. Document ID: JP 05279407 A

L31: Entry 8 of 10

File: JPAB

Oct 26, 1993

PUB-NO: JP405279407A
DOCUMENT-IDENTIFIER: JP 05279407 A
TITLE: PREPARATION OF CARBOXYLATED LATEX BY SELECTIVE MONOMER ADDITION AND
POLYMERIZATION

PUBN-DATE: October 26, 1993

INVENTOR-INFORMATION:

NAME

COUNTRY

PRENTICE, SUSAN M

GUJARATHI, RAMESH N

CHEN, SUN-LIN

INT-CL (IPC): C08F 2/22; C08F 2/00; C08F 2/44; C08F 212/06; C08F 220/00; C08F 236/04

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWIC

9. Document ID: EP 538722 A1

L31: Entry 9 of 10

File: EPAB

Apr 28, 1993

PUB-NO: EP000538722A1
DOCUMENT-IDENTIFIER: EP 538722 A1
TITLE: A process for the production of carboxylated latexes by the selective monomer
addition and polymerization.

PUBN-DATE: April 28, 1993

INVENTOR-INFORMATION:

NAME

COUNTRY

PRENTICE, SUSAN MONET

US

GUJARATHI, RAMESH N

US

CHEN, SUN-LIN NMN

US

INT-CL (IPC): C08F 212/04; C08F 236/10

EUR-CL (EPC): C08F279/02

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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KWIC

10. Document ID: EP 476528 A2

L31: Entry 10 of 10

File: EPAB

Mar 25, 1992

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 5,965,257
DATED : October 12, 1999
INVENTOR(S) : Younger Ahluwalia

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

[56] **References Cited:** Insert -- FOREIGN PATENT DOCUMENTS WO
82/00269A 2/1982.....WIPO --

[56] **References Cited:** Insert -- OTHER PUBLICATIONS DATABASE WPI
Section Ch, Week 8923, Derwent Publication Ltd., London, GB; Class A93,
AN90-171160 XP002079625 & RO 96 083 A (Trust Antr Gen Cons), November
10, 1988 --

Column 9,
Line 30, "ate" should be deleted
Line 55, "coatings" should read -- coating --

Signed and Sealed this

Eighteenth Day of September, 2001

Attest:

Nicholas P. Godici

Attesting Officer

NICHOLAS P. GODICI
Acting Director of the United States Patent and Trademark Office

PUB-NO: EP000476528A2
DOCUMENT-IDENTIFIER: EP 476528 A2
TITLE: Process for producing stable latex.

PUBN-DATE: March 25, 1992

INVENTOR-INFORMATION:

NAME	COUNTRY
CHEN, SUN-LIN	US
GUJARATHI, RAMESH N	US
PRENTICE, SUSAN MONET	US

US-CL-CURRENT: 524/145; 524/556
INT-CL (IPC): C08F 2/30; C08F 6/14; C08F 257/02
EUR-CL (EPC): C08F257/02

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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Term	Documents
CHEN.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	121258
CHENS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	4
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SUNS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1215
LIN[DWPI,EPAB,JPAB,USPT,PGPB]	72526
LINS[DWPI,EPAB,JPAB,USPT,PGPB]	1063
((CHEN ADJ SUN) ADJ (LIN[IN])).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	10
(CHEN, SUN LIN [IN]).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	10

[Display Format: -](#)[Change Format](#)[Previous Page](#)[Next Page](#)

United States Patent [19]
Bailey

[11] **Patent Number:** 4,954,545
[45] **Date of Patent:** Sep. 4, 1990

[54] **PROCESS FOR CLEANING POLYMER
PROCESSING EQUIPMENT**

[75] **Inventor:** Fay W. Bailey, Bartlesville, Okla.

[73] **Assignee:** Phillips Petroleum Company,
Bartlesville, Okla.

[21] **Appl. No.:** 283,350

[22] **Filed:** Dec. 12, 1988

Related U.S. Application Data

[62] **Division of Ser. No. 78,338, Jul. 27, 1987, Pat. No.
4,838,948.**

[51] **Int. Cl.³** C08K 3/26; C08K 3/32;
C08K 3/34; C11D 1/12

[52] **U.S. Cl.** 524/158; 524/157;
524/414; 524/425; 524/427; 524/442; 524/444;
252/121; 252/128

[58] **Field of Search** 524/158, 425, 414, 427,
524/442, 444, 157; 252/121, 128

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,661,622 5/1972 Rogers 117/75
4,386,169 5/1983 Artur et al. 524/161
4,456,710 6/1984 Lötters et al. 524/159

Primary Examiner—Allan M. Lieberman
Attorney, Agent, or Firm—Richmond, Phillips,
Hitchcock & Umphlett

[57] **ABSTRACT**

The present invention pertains to a process for cleaning polymeric processing equipment. The process comprises purging the polymer processing equipment with a composition comprising a polymer, a mild abrasive and a sulfonated surfactant.

12 Claims, 5 Drawing Sheets

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DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ

<u>L40</u>	silica near3 particle same width same length and rubber	15	<u>L40</u> no 3rd
<u>L39</u>	silica near3 particle same width same length	81	<u>L39</u>
<u>L38</u>	silica near3 particle near3 width near3 length	2	<u>L38</u>
<u>L37</u>	silica adj particle near width near length	0	<u>L37</u>
<u>L36</u>	Snowtex adj silicasol	0	<u>L36</u>
<u>L35</u>	Snowtex adj5 silicasol	4	<u>L35</u>
<u>L34</u>	Snowtex silicasol	0	<u>L34</u>
<u>L33</u>	Snowtex adj2 silicasol	2	<u>L33</u>
<u>L32</u>	snowtex silicasol	0	<u>L32</u>
<u>L31</u>	Chen, Sun Lin [in]	10	<u>L31</u>
<u>L30</u>	Colvin, Howard \$ [in]	36	<u>L30</u>
<u>L29</u>	L27 and (rubber or elastomer)	3	<u>L29</u>
<u>L28</u>	L27 and rubber	3	<u>L28</u>
<u>L27</u>	silica adj3 particle and width near3 (nm or nanometer) and length near3 (nm or nanometer)	20	<u>L27</u>
<u>L26</u>	silica adj particle and width near (nm or nanometer) and length near (nm or nanometer)	1	<u>L26</u>
<u>L25</u>	silica adj particle and width near (nm or nanometer)	52	<u>L25</u>
<u>L24</u>	width near (nm or nanometer)	1594	<u>L24</u>
<u>L23</u>	L21 and rubber [ti]	39	<u>L23</u>
<u>L22</u>	L21 and rubber	372	<u>L22</u>
<u>L21</u>	l1 near l19	2269	<u>L21</u>
<u>L20</u>	l1 and l19	201608	<u>L20</u>
<u>L19</u>	dimension or size	2813371	<u>L19</u>
<u>L18</u>	l1 near l2	140	<u>L18</u>
<u>L17</u>	l1 and l2	143476	<u>L17</u>
<u>L16</u>	l1 near l2 [ab]	17	<u>L16</u>
<u>L15</u>	l1 near l2 [ti]	0	<u>L15</u>
<u>L14</u>	l1 near l2	140	<u>L14</u>
<u>L13</u>	l1 with l2	4521	<u>L13</u>
<u>L12</u>	l1 same l2	15847	<u>L12</u>
<u>L11</u>	l7 and l1	0	<u>L11</u>
<u>L10</u>	l8 and l1	0	<u>L10</u>
<u>L9</u>	l2 near5 l3 near1 l4	0	<u>L9</u>
<u>L8</u>	l2 near5 l3 near2 l4	3	<u>L8</u>

acid, fumaric acid or itaconic acid and monohydric

the outside appearance, gloss and richness of a coating

compound having free isocyanate groups, such as a polyisocyanate compound, an alcoholate of a polyvalent metal, etc. are used as the curing agent (II), the use of the hydroxyl group-containing vinyl monomers is undesirable since it reduces the storage stability of the resulting composition as a one-can paint. On the other hand, when an aminoplast resin is used as the curing agent (II), the hydroxyl group-containing vinyl monomers can be used. In view of the fact that a curing catalyst is added optionally to impart low-temperature curability, the amount of the hydroxyl group-containing vinyl monomer used should be less than 5% by weight which amount does not adversely affect the storage stability of the composition as a one-can paint.

The vinyl polymer (I) may be obtained by conventional known methods using the monomers exemplified above. For example, the polymerization may be carried out by the solution polymerization technique using radical generators.

The radical generators may be any of those which can be used normally in the polymerization of acrylic monomers. Typical examples of the radical generators are azobisisobutyronitrile, di-tert-butyl peroxide and benzoyl peroxide. Examples of solvents that can be used in the polymerization include aromatic hydrocarbons such as toluene or xylene; acetic acid esters such as ethyl acetate, butyl acetate, or Cellosolve acetate; ketones such as methyl ethyl ketone or methyl isobutyl ketone; alcohols such as butanol or isobutanol; and Cellosolves such as methyl Cellosolve or ethyl Cellosolve. When a polyisocyanate compound having a free isocyanate group is used as the curing agent (II), alcohols should not be used as these solvents.

As required, there can be used ordinary chain transfer agents such as mercaptan, alpha-methylstyrene dimer or terpene hydrocarbon having the molecular formula $C_{10}H_{16}$.

The vinyl polymer (I) so obtained preferably has a number average molecular weight (\bar{M}_n) of 500 to 50,000. If the \bar{M}_n of the polymer (I) is less than 500, the properties of a coated film prepared from the resulting composition are not sufficient. If, in an attempt to provide sufficient film properties, the hydroxyl number of the polymer (I) after liberation of trialkylsiloxy groups, etc. is increased, a coated film obtained from the resulting composition becomes undesirably brittle. On the other hand, if \bar{M}_n exceeds 50,000, the handling properties of the resulting composition in coating and

low weight and containing no free isocyanate groups with the isocyanate groups or water; and isocyanates (including oligomers) having an isocyanurate group obtained by polymerizing the aforesaid diisocyanates with each other.

Copolymers of at least one vinyl monomer having both at least one polymerizable unsaturated bond and at least one isocyanate group per molecule, typified by isocyanatoethyl (meth)acrylate, isocyanatopropyl (meth)acrylate, isocyanatobutyl (meth)acrylate or isopropenyl isocyanate with at least one of vinyl monomers (B) (excepting the hydroxyl group-containing monomers) exemplified hereinabove may also be used as the polyisocyanate compound.

In view of the film properties to be obtained, the mixing ratio of the vinyl polymer (I) to the polyisocyanate compound is preferably such that the equivalent ratio of the hydroxyl groups generated from the polymer (I) to the isocyanate groups in the polyisocyanate compounds is from 1:0.2 to 1:3.

The polyisocyanate compound may be incorporated in the vinyl polymer (I) in advance to form a one-can paint, or may be stored separately from the vinyl polymer (I) and mixed with it just prior to coating (two-can paint).

When the vinyl polymer (I) is prepared by using the hydroxyl group-containing vinyl monomer as the vinyl monomer (B), an aminoplast is preferred as the curing agent (II) in view of the storage stability of the resulting composition, although the polyisocyanate compound, chelate compound or polyvalent alcoholate may be incorporated in the polymer (I) immediately prior to coating instead of using the aminoplast. The aminoplast is typically an aminoplast etherified with a monohydric alcohol, and is usually employed in this form. The presence of the alcohol in the coating resin composition of this invention is likely to lead to reaction of the alcohol with the siloxy group and to reduce the long-term stability of the composition. Hence, hexamethoxymethylmelamine not containing an alcohol is preferred.

The amount of the aminoplast used is preferably 10 to 40% by weight of the vinyl polymer (I). Where long-term storage stability is not required, an alcohol solution of an aminoplast etherified with a monohydric alcohol may be used as the curing agent (II).

The polyvalent metal alcoholate or chelate compound used as the curing agent (II) is typically an alcoholate or chelate compound of aluminum, titanium,

L7 l2 near5 l3 near5 l4
L6 l2 near5 l3 near l4
L5 l2 near l3 near l4
L4 ?0nm or ?0nanometer
L3 ?nm or ?nanometer
L2 (width or wide)
L1 silica or silicon dioxide

5 L7
0 L6
0 L5
6826 L4
9488 L3
2153543 L2
454892 L1

END OF SEARCH HISTORY

6

tyl phosphate tributaryethyl phosphate tristeryl phosphate

This catalyst is a siloxy group-dissociating catalyst (III) for hydrolyzing the siloxy groups in the vinyl polymer (I) to form hydroxyl groups in the polymer (I). 65 Examples of the dissociating catalyst (III) include phosphoric acid; phosphoric acid esters such as trimethyl phosphate, triethyl phosphate, tributyl phosphate, trioc-

Examples of the water-binding agent include trialkyl ortho-formates such as trimethyl ortho-formate, triethyl ortho-formate and tributyl ortho-formate; trialkyl ortho-acetates such as trimethyl ortho-acetate, triethyl ortho-acetate and tributyl ortho-acetate; trialkyl ortho-borates such as tributyl ortho-borate and triethyl ortho-borate; tetra(substituted)alkyl silicates such as tetramethyl silicate, tetraethyl silicate, tetrabutyl silicate, tetra(2-methoxyethyl) silicate and tetra(2-chloroethyl) silicate; equivalents of the above tetra(substituted)alkyl silicates such as tetraphenyl silicate and tetrabenzyl silicate; hydrolyzable ester compounds such as condensation products (e.g., dimers, trimers, tetramers, hexamers, etc.) of the above tetra(substituted)alkyl silicates or the equivalents thereof; compounds having an isocyanate group, such as phenyl isocyanate, p-chlorophenyl isocyanate, benzenesulfonyl isocyanate, p-toluenesulfonyl isocyanate and isocyanatoethyl methacrylate; acetals such as dimethoxyethane and 1,1-dimethoxypropane; and ketals such as 2,2-dimethoxypropane and 2,2-dimethoxybutane.

Set Name Query

side by side

Hit Count Set Name

result set

DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ

<u>L40</u>	silica near3 particle same width same length and rubber	15	<u>L40</u>
<u>L39</u>	silica near3 particle same width same length	81	<u>L39</u>
<u>L38</u>	silica near3 particle near3 width near3 length	2	<u>L38</u>
<u>L37</u>	silica adj particle near width near length	0	<u>L37</u>
<u>L36</u>	Snowtex adj silicasol	0	<u>L36</u>
<u>L35</u>	Snowtex adj5 silicasol	4	<u>L35</u>
<u>L34</u>	Snowtex silicasol	0	<u>L34</u>
<u>L33</u>	Snowtex adj2 silicasol	2	<u>L33</u>
<u>L32</u>	snowtex silicasol	0	<u>L32</u>
<u>L31</u>	Chen, Sun Lin [in]	10	<u>L31</u>
<u>L30</u>	Colvin, Howard \$ [in]	36	<u>L30</u>
<u>L29</u>	L27 and (rubber or elastomer)	3	<u>L29</u>
<u>L28</u>	L27 and rubber	3	<u>L28</u>
<u>L27</u>	silica adj3 particle and width near3 (nm or nanometer) and length near3 (nm or nanometer)	20	<u>L27</u>
<u>L26</u>	silica adj particle and width near (nm or nanometer) and length near (nm or nanometer)	1	<u>L26</u>
<u>L25</u>	silica adj particle and width near (nm or nanometer)	52	<u>L25</u>
<u>L24</u>	width near (nm or nanometer)	1594	<u>L24</u>
<u>L23</u>	L21 and rubber [ti]	39	<u>L23</u>
<u>L22</u>	L21 and rubber	372	<u>L22</u>
<u>L21</u>	l1 near l19	2269	<u>L21</u>
<u>L20</u>	l1 and l19	201608	<u>L20</u>
<u>L19</u>	dimension or size	2813371	<u>L19</u>
<u>L18</u>	l1 near l2	140	<u>L18</u>
<u>L17</u>	l1 and l2	143476	<u>L17</u>
<u>L16</u>	l1 near l2 [ab]	17	<u>L16</u>
<u>L15</u>	l1 near l2 [ti]	0	<u>L15</u>
<u>L14</u>	l1 near l2	140	<u>L14</u>
<u>L13</u>	l1 with l2	4521	<u>L13</u>
<u>L12</u>	l1 same l2	15847	<u>L12</u>
<u>L11</u>	l7 and l1	0	<u>L11</u>
<u>L10</u>	l8 and l1	0	<u>L10</u>
<u>L9</u>	l2 near5 l3 near1 l4	0	<u>L9</u>
<u>L8</u>	l2 near5 l3 near2 l4	3	<u>L8</u>

A resin solution having a non-volatile content of

over the course of 5 hours. The mixture was then
at the above temperature for 10 hours to give a resin
solution having a non-volatile content of 49.0%, a vis- 25
cosity of A, a number average molecular weight of 400
and an OH value of 24.

following meanings.

⊙: Very good

○: Good

△: Fair

×: Poor

TABLE I

Vinyl polymer used		E 1	E 2 RE 1	E 3	CE 1 RE 6	E 4	E 5 RE 2	E 6 RE 3	
Vinyl polymer solution		100	100	100	100	100	100	100	
Paint formation (parts)	"Barriock DN-950"	25		25	27	4.8			
	Hexamethoxymethylmelamine		36				21	36	
	p-Toluenesulfonic acid		0.5			1	0.3	0.7	
	Dibutyltin diacetate	0.1							
	Methacryloylethyl phosphate		0.1						
	Trimethyl ortho-formate	1						1	
	Tetraethyl silicate								
Properties	Rutile-type titanium oxide	46	48	46	47	36	42	48	
	Gloss (60°)	93	90	95	88	91	89	93	
	Hardness (pencil hardness)	H	2H	2H	2H	F	F	3H	
	Adhesion (cross hatch test)	100/100	100/100	100/100	100/100	100/100	100/100	100/100	
	Xylene resistance (number of rubbings)	60	78	75	55	40	53	70	
	Moisture resistance (50° C., 98% humidity, 240 hrs.)	⊙	⊙	⊙	⊙	⊙	⊙	⊙	
	Bending resistance (6 mm ϕ)	°	°	°	°	⊙	⊙	°	
	Storage stability (40° C., 1 month)	⊙	⊙	⊙	X	⊙	⊙	⊙	
	Remarks				Gelled in one day				

Vinyl polymer used		CE 2 RE 7	E 7 RE 4	E 8	E 9 RE 5	CE 3 RE 3	CE 4 RE 9	
Paint formation (parts)	Vinyl polymer solution "Barriock DN-950"	100	100	100	100	100	100	
	Hexamethoxymethylmelamine	36			36			
	p-Toluenesulfonic acid	0.5			0.5			
	Dibutyltin diacetate		0.1	0.5		0.1	0.1	
	Methacryloylethyl phosphate							
	Trimethyl ortho-formate							
	Tetraethyl silicate		1	1		2	1	
Properties	Rutile-type titanium oxide	48	39	44	48	34	39	
	Gloss (60°)	87	90	95	93	83	90	
	Hardness (pencil hardness)	3H	2H	H	H	2B	2B	
	Adhesion (cross hatch test)	100/100	100/100	100/100	100/100	50/100	30/100	
	Xylene resistance (number of rubbings)	73	51	63	69	5	3	
	Moisture resistance (50° C., 98% humidity, 240 hrs.)	⊙	⊙	⊙	⊙			
	Bending resistance (6 mm ϕ)	Δ	°	°	°	X	X	
	Storage stability (40° C., 1 month)	X	⊙	⊙	⊙	⊙	⊙	
	Remarks		Gelled					

<u>L7</u>	l2 near5 l3 near5 l4	5	<u>L7</u>
<u>L6</u>	l2 near5 l3 near l4	0	<u>L6</u>
<u>L5</u>	l2 near l3 near l4	0	<u>L5</u>
<u>L4</u>	?0nm or ?0nanometer	6826	<u>L4</u>
<u>L3</u>	?nm or ?nanometer	9488	<u>L3</u>
<u>L2</u>	(width or wide)	2153543	<u>L2</u>
<u>L1</u>	silica or silicon dioxide	454892	<u>L1</u>

END OF SEARCH HISTORY

- (II) a curing agent having reactivity with the hydroxyl groups generated upon reaction of the at least one siloxy group from the polymer (I) with moisture in the air, wherein said copolymerizable vinyl monomer is selected from the group consisting of (meth)acrylic acid esters containing no functional groups, aromatic vinyl monomers, hydroxyl group-containing vinyl monomers, (meth)acrylic acid and mixtures thereof.
2. A coating resin composition comprising:
- (I) a vinyl polymer obtained by polymerizing (A) 5 to 90% by weight of a vinyl monomer having at least one unsaturated double bond and at least one siloxy group per molecule whereby upon reaction with moisture in the air, the siloxy group forms a hydroxyl group and (B) 95 to 10% by weight of a copolymerizable vinyl monomer other than (A) in the presence of a radical generator,
- (II) a curing agent having reactivity with the hydroxyl groups generated upon reaction of the at least one siloxy group from the polymer (I) with moisture in the air, and
- (III) a siloxy group—dissociating catalyst, wherein said copolymerizable vinyl monomer is selected from the group consisting of (meth)acrylic acid esters containing no functional groups, aromatic vinyl monomers, hydroxyl group-containing vinyl monomers, (meth)acrylic acid and mixtures thereof.
3. The coating resin composition of claim 1 or 2 wherein the vinyl monomer (A) is a compound having both at least one unsaturated double bond and at least one siloxy group bonded to the carbon atom represented by the following formula



wherein R_1 and R_2 are identical or different and each represents a phenyl, allyl or C_1 - C_{18} alkyl group or a hydrogen, chlorine or fluorine atom, and R_3 represents a phenyl, allyl or C_1 - C_{18} alkyl group, per molecule.

the dissociating catalyst (III) is a metal salt of a siloxy group.

10. The coating resin composition of claim 2 wherein the dissociating catalyst (III) is a metal salt of a siloxy group.

11. The coating resin composition of claim 2 wherein the dissociating catalyst (III) is an organic tin compound.

12. The coating resin composition of claim 2 wherein the dissociating catalyst (III) is a compound capable of yielding a fluorine ion.

13. The coating resin composition of claim 4 wherein the amount of the polyisocyanate compound is such that the equivalent ratio of the hydroxyl group generated from the siloxy groups of the vinyl polymer (I) to the isocyanate groups in the polyisocyanate compound is from 1:0.2 to 1:3.

14. The coating resin composition of claim 4 wherein the polyisocyanate compound is an aromatic diisocyanate, an aliphatic diisocyanate, an alicyclic diisocyanate or an adduct of said diisocyanate compound with a polyhydric alcohol, a polyester resin or water.

15. The coating resin composition of claim 4 wherein the polyisocyanate compound is an isocyanate having an isocyanurate group obtained by polymerizing an aromatic diisocyanate, an aliphatic diisocyanate or an alicyclic diisocyanate.

16. The coating resin composition of claim 4 wherein the polyisocyanate compound is a copolymer of at least one vinyl monomer having both at least one polymerizable unsaturated bond and at least one isocyanate group per molecule with another copolymerizable vinyl monomer not containing a hydroxyl group.

17. The coating resin composition of claim 1 or 2 wherein said copolymerizable vinyl monomer is selected from the group consisting of C_1 - C_{22} alkyl(meth)acrylates; 2-ethoxyethyl (meth)acrylates; cyclohexyl (meth)acrylate; styrene; vinyl toluene; t-butylstyrene; alpha-methylstyrene; hydroxyethyl vinyl ether; hydroxybutyl vinyl ether; beta-hydroxyethyl (meth)acrylate; beta-hydroxypropyl (meth)acrylate; beta-hydroxybutyl (meth)acrylate; N-methylolated (meth)acrylamide; adduct of beta-hydroxyethyl (meth)acrylate with epsilon-caprolactone; polyethylene glycol mono (meth)acrylate; polypropylene glycol mono (meth)acrylate; adducts of (meth)acrylic acid, maleic acid, fumaric acid or itaconic acid with monoglycidyl esters of branched fatty acids, glycidyl octylate, glycidyl ester of coconut